

AD 682216

ONR Contract No. Nonr 591-(10)
R.P.I. Project No. 441.35

Technical Report No. 34
November, 1968

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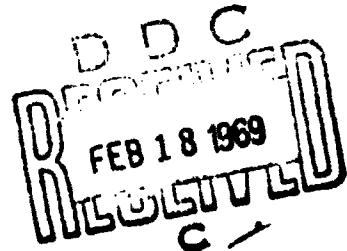
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Submitted to Chemical Instrumentation for publication

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ABSTRACT

Some 200 surface tension determinations have been made on 107 single-salt melts using eight experimental techniques. From a consideration of the experimental difficulties, such as the inconvenience of visual observation on the melt, the corrosive nature of the melt, volatilization and condensation of salt vapors on apparatus, the most versatile method recognized to be applicable to these molten systems at elevated temperatures is the method of Maximum Bubble Pressure, by which 75 per cent of the total determinations on the 107 salts were made. Other methods in descending percentages of application are: Wilhelmy Slide Plate, Capillary Rise, Maximum Pull on Cylinder, Pin Method, Pendant Drop, Ring Method and Sessile Bubble. The basic principles, surface tension range of applicability and temperature limitation for these techniques are briefly considered.

INTRODUCTION

There is much information relatively at large in the literature relative to the surface tension of inorganic compounds in molten state, but a careful and critical evaluation of the results and of experimental techniques in this area of high temperature chemistry appear an outstanding need. Eight experimental techniques, i.e., Maximum Bubble Pressure, Wilhelmy Slide Plate, Pin, Maximum Pull on Cylinder, Pendent Drop, Ring, Capillary Rise, Sessile bubble have been used to gain surface tension data of some 107 single-salt melts over a wide range of temperature. This article develops a review of the basic principles, experimental techniques, temperature limitations and other salient features for each method. Comparisons of the various methods are considered from the viewpoints of experimental difficulties, range of applicability and replicability of the results.

SURFACE TENSION TECHNIQUES FOR MOLTEN SALTS

(1) Maximum Bubble Pressure Method

The maximum bubble pressure method of determining the surface tension of a liquid, first suggested by Simon (31), was developed and applied to molten salt systems by Jaeger (20). It involves the very slow formation of a bubble at the tip of a capillary immersed in

the melt and the subsequent determination of the maximum pressure in the bubble at the very instant it bursts. Cantor (6) discussed the theory of bubble formation; his equation for the maximum bubble pressure, corrected by Schrödinger (29), is:

$$\gamma = \frac{pr}{2} \left[1 - \frac{2r}{3h} - \frac{1}{6} \left(\frac{r}{h} \right)^2 \dots \right] \quad (1)$$

where γ = surface tension of the melt, dyne cm^{-1}

r = radius of the capillary, cm

p = maximum pressure difference between the inside and outside of the bubble at the level of the end of the tip, dyne cm^{-2} .

$h = \frac{P}{g} / (p - p')$, the height in cm. of a column of the melt equivalent to pressure p

ρ = density of the melt being measured, gm. cm^{-3}

ρ' = density of the bubbling gas saturated with the salt vapor, gm. cm^{-3}

g = acceleration of gravity

Sugden (32) showed that the Schrödinger approximation is valid for values of $\frac{r}{h} < 0.2$. When $\frac{r}{h}$ exceeds 0.2, it becomes necessary to use the Sugden tables and his method of successive approximations (16). A thorough investigation of the maximum bubble pressure method by Hoffman (19) and Tripp (33) showed that the mathematical theory is in accord with experiment only if the bubbles are formed slowly (one bubble per 30 or 60 seconds). The phenomena are more complex and not readily amenable to theoretical treatment when the rate of bubble formation is high.

The most extensive application of this method has been by Jaeger (20) (51 salts) and by Ellis et al (8-13) (20 salts). The method appears well suited for molten salts having surface tensions ranging from 50 to 150 dyne cm⁻¹ and for the temperature interval from room temperature to 1600°C.

The method is not readily applicable to viscous melts (e.g. ZnCl₂) and highly volatile systems (7). Irregular formation and behavior of the bubble can occur in melts of this type; the bubble may burst into a number of smaller bubbles with a resultant stepwise pressure drop in the system; alternatively the bubble may not burst despite a rise and fall of pressure, or the pressure response when the bubble bursts may be slow and abnormally small.

Some of the experimental features that distinguish the high-temperature application of this method to inorganic molten salts from the ambient-temperature applications to aqueous and organic liquids are as follows:

(a) Since certain melts may be quite corrosive, the radius of the capillary tip should be checked, preferably after each experiment. In addition, the time and area of contact of the melt with various components of the surface tension assembly should be minimized.

(b) Since visual observations of the melt are frequently not possible, the contact of the capillary tip with the surface of the melt must be detected by indirect techniques (e.g. electrical contact).

(c) To prevent a possible cooling effect at the surface of the melt during the formation and release of the bubbles from the capillary

tip, preheating of the inert bubbling gas to the temperature of the measurement was recommended by Jaeger (20) and Semenchenko and Shikhobalova (30). For very slow bubbling rates, Dahl and Duke (7) found this step unnecessary.

(ii) Detachment Methods

The term "detachment method" denotes any method of surface tension determination which is based on a measurement of the force required to detach an object from the surface of the liquid. The maximum pull just before the object is detached from the surface will be equal to the weight of the object plus γL , where L is the total perimeter introduced into the melt. This assumes that the weight of the liquid which has been raised above the liquid surface, at the moment of detachment, is given by the total tension that its surface will support and that the contact angle is zero. The following four methods may be classified as molten salt surface tension detachment methods.

(a) Wilhelmy Slide Plate Method

The simplest application of the above principle is that of Wilhelmy (34). In this method the maximum pulling force necessary to detach a thin platinum plate from the liquid surface is determined. For a straight edge, the force is proportional to the surface tension of the liquid. The plate, suspended from one arm of a balance, is dipped into the liquid and the melt container is lowered until detachment occurs. The "pull" on the balance arm is noted at the instant detachment is effected. The surface tension is calculated by the expression:

$$W_{\text{tot}} = W_{\text{slide}} + 2\gamma(L+x) \quad (2)$$

where L = width of the slide

x = the thickness of the slide

W_{tot} , W_{slide} = the observed weights at the moment of detachment and before dipping into liquid respectively.

End effects are assumed negligible. This relatively simple method has been estimated to be accurate to within $\pm 0.1\%$ for room temperature measurements of aqueous and organic liquids (28), provided that the liquid wets the slide. Bertozzi (2,3) applied this technique to molten alkali metal nitrates (300 - 600°C) and halides (600 - 900°C) with an accuracy of $\pm 0.6\%$.

(b) Pin Method

This procedure was introduced by Janz and Lorenz (21,22) to measure simultaneously the surface tensions and densities of molten alkali nitrates and carbonates, and has been used by Morris, McNair and Koops (25) for the molten molybdates. In this method, a surface tension detachment pin is machined to be part of an Archimedean density bob, which is suspended from an analytical balance. The weight is noted at room temperature and again at the working temperature after temperature equilibrium has been reached in order to correct for the slightly decreased buoyant force of air at higher temperatures. The crucible with the melt is then raised until the surface tension pin just contacts the melt; at this point the contact weight (W_c) and the relative crucible height are noted. The crucible is then lowered and the maximum "pull", just at the moment of detachment, is measured. The break-point weight (W_b) and the final rest weight (W_r)

are thus obtained.

The surface tension pull on the bob is $2\pi r\gamma$ dynes, where r and γ have their usual significance; this is equal to the difference between the break and the rest weights,

$$gW_d = g(W_b - W_r)$$

assuming zero angle of contact at the break-point. Thus, it follows that the surface tension is given by:

$$\gamma = \frac{gW_d}{2\pi r} = AW_d \quad (3)$$

where the constant A is a characteristic of the pin dimensions. The radius of the pin at any temperature $t^\circ C$ may be calculated from the relations:

$$r = r_0 (1 + a\Delta t) \quad (4)$$

where r_0 = radius of the pin at the calibration temperature (t_0),
 a = the coefficient of linear expansion of the pin material and
 $\Delta t = (t - t_0)$. Hence, the final form of the surface tension equation is:

$$\gamma = \frac{A_0 W_d}{1 + a\Delta t} \quad (5)$$

where $A_0 = \frac{g}{2\pi r_0}$. The constant A_0 may be calculated directly from the measurement of r_0 or obtained indirectly from measurements with calibration liquids of accurately known surface tensions.

The pin method, although has not been theoretically justified, has been verified experimentally in the case of molten Li_2CO_3 , the surface tension of which has been measured independently by Jenz and Lorenz (22) using the pin method and by Moiseev and Stepanov (24b) using the maximum bubble pressure technique. Both sets of results agree to within 0.3% in the temperature range studied.

(c) Maximum Pull on Cylinder Method

This method is a modification of the ring method in which the ring is replaced by a vertical hollow cylinder and has been widely used both at ambient and at elevated temperatures. An expression similar to that developed by Harkins, Young and Cheng (18) for the ring method is used to calculate the surface tension, i.e.

$$\gamma = \frac{Mg}{4\pi R} F \quad (6)$$

where R = the mean radius of the cylinder, cm

F = a dimensionless factor depending on R^3/V and R/t

V = volume of liquid held up by the cylinder ($= M/\rho_1$) cm^3

t = the thickness of the cylinder wall, cm.

King (23), using this method for determining the surface tensions of silicate slags, found that the F -factors did not have the same values for a cylinder and for a ring. Using liquids of known surface tensions, he determined correction factors and hence a calibration curve applicable to the cylinder ($R = \frac{1}{4}$ inch). Bradbury and Maddocks (4), also using a cylinder of $R = \frac{1}{4}$ inch, obtained a somewhat different calibration for $F(R^3/V)$ versus R^3/V . No explanation was advanced for the difference in the two calibration curves although cylinders of the same R -value were used. The above difference may be attributed, in part, to the neglect of the dimensionless variable R/t in the preceding calibrations and to possible differences in the thickness of the walls of the cylinders used by King (23) and by Bradbury and Maddocks (4).

The effect of the contact angle, θ , on the maximum pull on the cylinder was investigated by King and was shown to be negligible unless $\theta > 40^\circ$.

(d) Ring Method

The theoretical principles of this method were established by Harkins, Young and Cheng (18), and further refined by Harkins and Jordan (17). A complete discussion of the theory and experimental aspects of this method at ambient temperature is given by Harkins (16). This method has been used at high temperatures by Callis et al (5) for sodium metaphosphate and mixtures of Na_2O and P_2O_5 , with due cognizance to the correction factors of Harkins and Jordan (17) for the calculation of surface tensions.

(iii) Capillary Rise Method

The capillary rise method is the simplest method to operate in a closed system, a requirement for low-melting salts with high volatility (e.g. UF_6 (26), ZnCl_2 (12), GaCl_3 (14,15) and its organic complexes). The theoretical principles of this method are well established; it is best suited for systems where visual observation of the melt is possible. For a single capillary, Harkins (16) gives:

$$\gamma = \frac{1}{2}rhg(\rho - \rho') \quad (7)$$

where γ = surface tension, dyne cm^{-1}

r = radius of capillary, cm

h = height of meniscus from immersed end of capillary, cm

g = acceleration of gravity

ρ = density of liquid measured, gm. cm⁻³

ρ' = density of vapor, gm. cm⁻³

For double capillary, Sudgen (32) gives:

$$\gamma = \frac{1}{2}H(\rho - \rho') g \left(\frac{1}{b_1} - \frac{1}{b_2} \right)^{-1} \quad (8)$$

where γ , g , ρ , ρ' carry the same significance as above, H is the vertical distance between the lowest points of the menisci in the two vertical tubes of radius r_1 and r_2 , cm, and b_1 and b_2 are the radii of curvature at these points, cm.

To use equation 8 for the calculation of surface tension, it is necessary to have some relation between the radius of the tube and the radius of curvature at the bottom of the meniscus (32).

The derivations of eqs. 7 and 8 assume that angle of contact is zero. This fact was experimentally verified only in cases of water- and some organic-glass systems by Richards and Carver (27), and may not be valid in cases of molten salts in contact with the apparatus material.

(iv) Methods Based on the Shape of Static Drops or Bubbles.

The general procedure for this class of methods is to form a liquid drop or a gas bubble in the liquid studied under conditions such that it is not subject to disturbance, and then to make certain measurements of its dimensions, e.g. from a photograph. These methods favor the observations of long term changes in surface tensions i.e. if very slow time effects are involved.

(a) Sessile Bubble Method

This technique consists of mounting a square-ended tube vertically

in a square spectrometer cuvette containing the liquid. A bubble of inert gas is grown at the end of the tube and held stationary there while a photograph is made with a microphotographic camera. The equatorial diameter and height of the bubble are then measured in the print by use of a travelling microscope. The magnification factor can be checked by measuring the diameter of the tube, which is shown in the photograph.

This method has only been applied to one molten salt system-ZnCl₂ (13). The author calculated the surface tension of ZnCl₂ from the dimensions of the bubble by use of Bashforth and Adams Tables (16). The results were claimed to be of low precision because of difficulties in measuring the pertinent dimensions. No further application of this method to molten salt systems is to be expected due to this fact.

(b) Pendant Drop Method

In this method, the salt is melted to form a drop hanging on the end of an inert rod. Surface tension can be calculated from measurements of the absolute drop diameter and a shape factor fixed by a "selected" diameter, d_s, defined as the diameter normal to the drop axis at a distance from the base of the drop equal to the maximum diameter, d_m of the drop. The surface tension is related to the liquid density, the absolute value of d_m and the ratio $\frac{d_s}{d_m}$ as given by Harkins (16).

This method has only been applied to inorganic melts in the case of oxide systems (24) of Al₂O₃, B₂O₃, GeO₂, SiO₂ and P₂O₅ with an overall accuracy of no better than $\pm 7\%$.

DISCUSSION

The preceding considerations of the various techniques, temperature range, the surface tension range and equations are summarized in Table 1. The fraction of the total determinations made by each technique is tabulated in Table 2 as the percent application. The surface tension range and temperature range of application are given in Fig. 1 as a guide relative to the applicability of each technique.

A comparison of the results gained by various techniques is of interest. For NaCl, eight independent investigations have been reported using three different techniques i.e., the maximum bubble pressure method, (3a,7a,20,24a,30,31a) the plate method (3) and the ring method (1a). These results are represented in Fig. 2. The insert in Fig. 2 illustrates an alternate presentation in which the percent departure of the various results from the values of Sokolova and Voskresenskaya (31a) are shown; the results of Sokolova and Voskresenskaya were selected as the reference norm from a consideration of such factors as preparation, purity of the salt, temperature range of study as well as precision and accuracy of the results. Inspection shows that the values obtained by the maximum bubble pressure method (7a,20,31a,24a) and the ring method (1a) are in good agreement (± 0.5 dynes cm^{-1}); the results gained by the plate method (3) are consistently higher (3 to 4 dynes cm^{-1}). Relative to the surface

tension studies generally the maximum bubble pressure method is most widely used technique; undoubtedly because it has many advantages from the viewpoints of experimental difficulties such as the inconvenience of visual observation of the melts, the corrosive nature of the melts, volatilization and condensation of the salt vapors on the apparatus and thermal gradient effects. Moreover highly reliable and accurate results can be gained by this technique especially for melts in the range 50-150 dyne cm⁻¹.

ACKNOWLEDGMENT

This work was made possible, in large part, by the financial support received from the U.S. National Bureau of Standards, Office of Standard Reference Data, Washington, D.C.

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Table 1 Molten Salt Surface Tension Techniques: Theory and Practice

| Method | Temperature Range (°C) | Surface Tension Range (dynes cm ⁻¹) | Equations |
|-------------------------|---------------------------|---|---|
| Maximum Bubble Pressure | ambient - 1600 | 50 - 150 | $\gamma = \frac{\rho r}{2} \left[1 - \frac{2\pi}{3h} - \frac{1}{6} \left(\frac{r}{h} \right)^2 \dots \right]$ |
| Capillary Rise | ambient - 200 | 10 - 50 | Single cap. $\gamma = \frac{1}{2} rhg(\rho - \rho')$ |
| | | | Double cap. $\gamma = \frac{1}{2} rhg(\rho - \rho') \left(\frac{1}{b_1} - \frac{1}{b_2} \right)^{-1}$ |
| Plate | 500 - 900 | 50 - 150 | $W_{tot} = W_{plate} + 2\gamma(L + \epsilon)$ |
| Pin | 350 - 1100 | 150 - 250 | $\gamma = \frac{A_e W_d}{1 + \frac{W_d}{\rho A_e}}; A_e = \frac{g}{2\pi r}$ |
| Pendant Drop | 100 - 2000 | 150 - 700 | Apply $\frac{d_s}{d_m} \cdot d_m$, ρ to tables (16) |
| Cylinder | 800 - 1400 | 50 - 150 | $\gamma = \frac{Mg}{4\pi R} P_{cyl.}$ |
| Ring | 650 - 950 | 50 - 150 | $\gamma = \frac{Mg}{4\pi R} P_{ring}$ |
| Sessile Bubble | 350 - 500 | 10 - 50 | Use Bassforth and Adams Table: (16) |

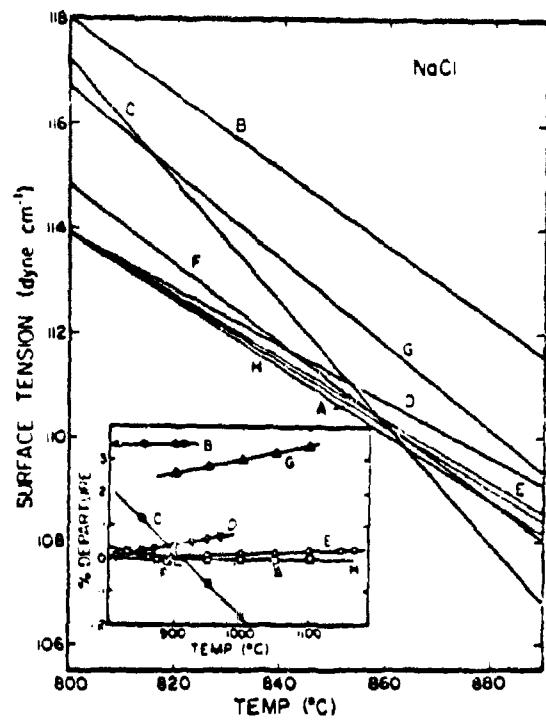
Table 2 Classification of the Molten Salt Surface Tension Techniques by the Fraction of the Total Determinations Made by Each Technique as Per Cent Application.

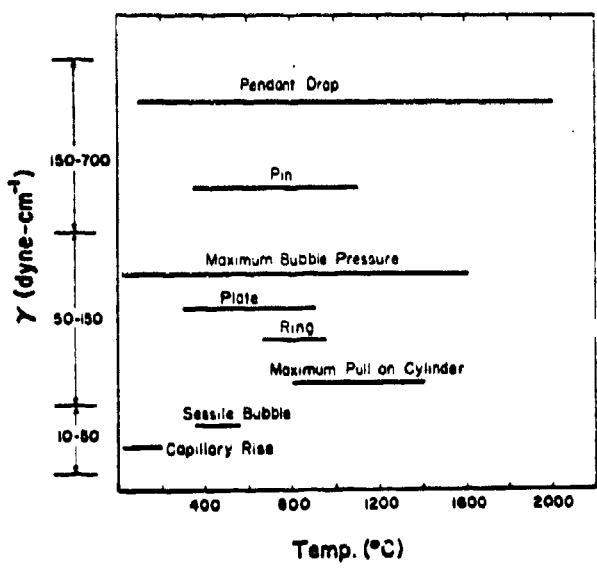
| Method | Per Cent Application |
|--------------------------|----------------------|
| Maximum bubble pressure | 74.6 |
| Wilhelmy slide plate | 7.2 |
| Capillary rise | 5.7 |
| Maximum pull on cylinder | 4.2 |
| Pin method | 3.2 |
| Pendant drop | 2.5 |
| Ring method | 2.0 |
| Sessile bubble | 0.6 |

Figure Caption Sheet

Fig. 1 Surface tension range and temperature range for the various techniques.

Fig. 2 Surface tension of molten NaCl obtained by the various techniques in the range 800-900°C: A, Barzakovskii (Ring, 1940)^{1a}; B, Bartozzi (Plate, 1965)³; C, Bloom, Davis and James (M.B.P., 1960)^{3a}; D, Desyatnikov (M.B.P., 1956)^{7a}; E, Jaeger (M.B.P., 1917)²⁰; F, Lantratov (M.B.P., 1961)^{24a}; G, Semenchenko (M.B.P., 1947)³⁰; H, Sokolova and Voskresenskaya (M.B.P., 1962).^{31a}





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| Rensselaer Polytechnic Institute | | Unclassified 2a GROUP | |
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| Surface Tension Techniques For Molten Salts | | | |
| 4. DESCRIPTIVE NOTES (Type of report and inclusive dates) | | | |
| Technical Report | | | |
| 5. AUTHOR(S) (Last name, first name, initial) | | | |
| G.J. Janz, J. Wong and G.R. Lakshminarayanan | | | |
| 6. REPORT DATE | 7a. TOTAL NO OF PAGES | 7b. NO. OF REPS | |
| November, 1968 | 18 | 34 | |
| 8a. CONTRACT OR GRANT NO. | 9a. ORIGINATOR'S REPORT NUMBER(S) | | |
| ONR Contract No. Nonr 591-(10) | Technical Report No. 34 | | |
| b. PROJECT NO. | | | |
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DD FORM 1 JAN 64 1473

Unclassified
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| 14. KEY WORDS | LINK A | | LINK B | | LINK C | |
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